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(54) Title: QUARTZ GLASS TUBE FOR USE IN THE PRODUCTION OF OPTICAL FIBER PREFORMS

(57) Abstract

An object of the present invention is to provide a quartz glass tube for optical fiber preforms capable of producing optical fiber preforms having high dimensional precision, excellent distribution of refraction index, and yet free from degradation and the like of working environment. The object above is accomplished by a quartz glass tube for use in the production of optical fiber preforms, characterized in that the high temperature viscosity thereof is varied in the wall thickness direction, and that the high temperature viscosity in the inner layer side is lower than that of the outer layer side. It is shown how the viscosity and the refractive index are influenced by different doping agents to optimize producing parameters for the optical fiber preform and the distribution of refraction index of the optical fiber preform obtained. A method for producing an optical fiber preform comprising inserting a core glass rod into said quartz glass tube and melt welding them together is also disclosed.

Quartz Glass Tube for Use in the Production of Optical Fiber Preforms

Detailed Description of the Invention

Industrial Field of Application

The present invention relates to a quartz glass tube for use in the production of optical fiber preforms and to an optical fiber preform using said quartz glass tube. In further detail, it relates to a quartz glass tube for optical fiber preforms capable of producing high-quality optical fibers with high productivity and at a low cost, and to a method for producing an optical fiber preform using said quartz glass tube for preforms. In a still further detail, it relates to a quartz glass tube for optical fiber preforms capable of producing a low-loss, high-quality optical fibers at a low cost.

Prior Art

Recently, larger amount of optical fibers, particularly, the single mode optical fibers, are being used with increasing practical use of optical fibers. However, it is expected that a still larger amount of optical fibers will be necessary with the expansion in its field of usage ranging from long distance main communication lines to domestic lines. To meet with such a demand in the expansion of usage, it is indispensable to realize mass production and cost reduction in the

production of optical fibers. This can be accomplished most simply by forming a large optical fiber preform and by then drawing it. In the conventional practical method for producing optical fiber preforms such as the vapor-phase axial deposition process (VAD process) or the outer vapor-phase deposition process (OVD process), the core portion and the clad portion are all produced by VAD or OVD process. Thus, if it is attempted to scale up or to obtain longer optical fiber preforms, there is a disadvantage that the production cost is increased in relation with the starting material, combustion gas, production facility, etc. Furthermore, if it is tried to form a further scaled up or a longer optical fiber preform, it is required that the soot body (i.e., a porous body obtained by depositing fine silica particles, which is a silica body obtained before vitrifying it into a transparent body, which is referred to herein as „a porous soot body") corresponding to the precursor for an optical fiber preform should be scaled up. Thus, if it is tried to form the po-rous soot body itself larger, there may occur troubles such as the generation of cracks or the drop off of the porous soot body, which may greatly lower the productivity. As a method for producing an optical fiber which overcomes the problems above, in JP-A-7-109136 (the term „JP-A-„ as referred herein signifies „an unexamined published Japanese patent application) and the like is proposed a production method, i.e., the so-called rod in tube method, which compri-ses forming the quartz glass tube for use as the clad portion accounting for 80 % or more of the cross section area by a method capable of forming a high performance quartz glass tube and yet, of reducing cost, and then monolithically integrating the resulting quartz glass tube with the core glass rod formed by, for example, the VAD process or the OVD method.

In addition to the mass production and the cost reduction mentioned above, low loss optical fibers are attracting attention these days, and, for example, there is proposed an optical fiber preform comprising a core portion prepared from a quartz glass doped with germanium and a clad portion formed by a quartz glass doped with chlorine or fluorine.

Problems the Invention is to Solve

The rod in tube method described above is a method for optical fiber preforms which easily enables a large scale and long optical fiber preforms and which is most suitable for mass production and lowering cost, however, for further cost reduction and increasing efficiency, a still yet improvement is required in order to more favorably realize melt welding of the clad tube with the core glass rod to obtain a monolithic body. For instance, in the rod in tube method, the core glass rod for use in the optical fiber preforms is subjected to melt welding into a monolithic body in a heating furnace in such a state that it is inserted into a quartz glass tube for optical fiber preforms. Thus, it happens that the quartz glass tube for the preforms above is heated to

a higher temperature on the outer side as compared to the inner side, and such a distribution in temperature brings about problems. That is, because the temperature distribution is as such that the inner side farther from the heating source becomes lower, if the inner side to be melt welded with the core rod for use in preforms above is set at a temperature sufficiently high as to cause melting, the outer side is heated to an excessively high temperature as to cause deformation, and this leads to the deterioration of dimensional precision. On the contrary, if the inner side is set at a low heating temperature as such to suppress the melt deformation of the outer side, the melting of the inner side becomes insufficient as to result in an insufficient melting of the inner side, thereby causing a fear that a favorable melt welding of the core glass for preforms can not be performed to obtain a monolithic body. Thus, in case of producing the optical fiber preform in accordance with the aforementioned rod in tube method by forming the core portion with a germanium doped quartz glass and the clad portion with a chlorine doped quartz glass, there is found a problem of the high temperature viscosity of the quartz glass tube for preforms in addition to the problem of the difference in refraction index. That is, in case of using a quartz glass tube doped with chlorine alone over the entire tube for the clad (or the over clad), although dehydration effect can be achieved over the entire tube, the refraction index of the quartz glass tube increases attributed to chlorine, thereby decreasing the difference in relative refraction index. Thus, to obtain a desired difference in relative refraction index, a further larger amount of germanium must be doped to further increase the refraction index of the core side, and this hinders the cost reduction and the increase of efficiency. Moreover, because the quartz glass tube for preforms doped with chlorine alone yields a higher viscosity as compared with a quartz glass doped with fluorine alone, the temperature of the heating furnace must be set at a temperature as high as such in a range of from 2000 to 2500°C in order to sufficiently perform the melt welding thereof with the core glass rod for preforms. This not only increases the cost of producing the heating furnace, but also impairs the working environment. In case chlorine is doped into the entire quartz glass tube for preforms, the problem of temperature distribution differing in the inner and outer side of the quartz glas tube and the core glass tube occurs similar to the case of doping only fluorine. Moreover, if melt welding for producing the monolithic body is carried out by heating the quartz glass tube and the core glass rod at such a high temperature above, germanium doped into the core may diffuse by the heat as to change the distribution in refraction index of the core portion, or the germanium diffused into the clad portion may change the distribution of refraction index inside the clad portion itself, and hence, it is feared that an optical fiber preform having favorable distribution in refraction index may not be obtained. Furthermore, in case of melt welding the quartz glass tube for preforms with the core glass rod, if the temperature is set as high as to sufficiently cause melting, the temperature of

the outer side of the quartz glass tube as above would be excessively increased as to cause a deformation due to melting, and there remains a problem as such that it would impair the dimensional precision of the outer diameter of the thus obtained optical fiber preform. Similarly, if the heating temperature should be set as low as such that the melt deformation of the outer side of the tube is suppressed, the inner side undergoes insufficient melting, and it is therefore difficult to solve the problem of causing a disadvantage as such that a favorable melt welding of the quartz glass tube with the core glass rod cannot be realized in obtaining a monolithic body. In the case of using a quartz glass tube for preforms doped with only fluorine, on the other hand, similar to the case of doping chlorine alone, not only the dehydration function can be obtained but the refraction index of the quartz glass tube decreases, and thereby a large difference in relative refraction index can be obtained. Furthermore, the high temperature viscosity in this case can be greatly decreased as compared with the case of doping chlorine. However, in case of doping fluorine over the entire quartz glass, the drop in viscosity occurs similarly on the inner side and the outer side in the direction of wall thickness. Thus, similar to the case of doping chlorine alone to the quartz glass tube, the problem of causing temperature distribution in the inner and the outer side remains unsolved.

In the light of the circumstances above, it has been found that a quartz glass tube for optical fiber preforms free from the aforementioned disadvantages can be obtained varying the high temperature viscosity of the quartz glass tube for optical fiber preforms in the wall thickness direction, and by lowering the high temperature viscosity of the inner layer side as compared with that of outer layer side. The present invention has been accomplished based on the above findings.

Furthermore, another object of the present invention is to provide an optical fiber preform capable for producing a low loss optical fiber. It is obtained by melt welding the quartz glass tube for optical fiber preforms above with a core rod for optical fiber preforms into a monolithic body.

Means for Solving the Problems

The present invention which accomplishes the object above provides a quartz glass tube for use in the production of optical fiber preforms, characterized in that the high temperature viscosity of the quartz glass tube is varied in the wall thickness direction, and that the high tem-

perature viscosity in the inner layer side is lower than that of the outer layer side, and an optical fiber preform using said quartz glass tube for optical fiber preforms.

The quartz glass tube for optical fiber preforms above is, as described above, a high purity quartz glass tube for optical fiber preforms, in which the high temperature viscosity thereof is varied in the wall thickness direction, and that the high temperature viscosity in the inner layer side is lower than that of the outer layer side. Preferably, the viscosity of the inner layer side at 1280 °C is in the range of from 1×10^4 to 1×10^{12} Poise, preferably in the range of from 1×10^{10} to 1×10^{12} Poise, and the viscosity of the outer layer side at 1280°C is in the range of from 1×10^{11} to 1×10^{13} Poise. The quartz glass tube can be produced by a conventionally employed practical methods such as the VAD method, OVD method, or the MCVD method, and can be produced from, for instance, a quartz glass ingot obtained by vitrifying, into a transparent body, a porous silica body prepared by hydrolyzing silicon compound (such as silicon tetrachloride or a siloxane compound), or by vitrifying by Verneuil's method, into a transparent body, a powder of rock crystal prepared by pulverizing and purifying a naturally occurring rock crystal, or a synthetic quartz glass ingot obtained by a sol-gel process, etc. In such cases, it is essential that the quartz glass tube is such the high temperature viscosity thereof is varied in the wall thickness direction, and that the high temperature viscosity in the inner layer side is lower than that of the outer layer side. In the preparation of the inner layer side and the outer layer side, it is preferred to dope a doping agent to either of the inner and the outer sides, or to both of them. As the doping agent, specifically mentioned are chlorine, germanium, phosphorus, and fluorine, which are used either alone or in combination of two or more selected therefrom. Preferably, at least one selected from chlorine, germanium, phosphorus, and fluorine is doped into the inner layer side of the quartz glass tube. More preferably, at least one selected from chlorine, germanium, and phosphorus is doped together with fluorine into the inner layer side, while doping chlorine into the outer layer side. By producing an optical fiber preform using the quartz glass tube for the optical fiber preform, the temperature at the melt welding can be set low, and thereby the melt welding in preparing the monolithic body can be performed while maintaining the dimensional precision of the outer diameter at a high level, and yet, while suppressing the deterioration of the working environment at high temperatures. Furthermore, the production cost of the heating furnace for use in the melt welding to obtain a monolithic body can be suppressed, and an optical fiber preform having the desired difference in relative refraction index can be obtained while preventing mal distribution in refraction index attributed to the diffusion of doping agents from occurring.

As a specific production method for obtaining the quartz glass tube for optical fiber pre-forms above, there can be mentioned, for instance: (1) an example of employing OVD method, which comprises forming a soot body by depositing fine silica glass particles generated by flame hydrolysis of high purity silicon tetrachloride on a heat-resistant base body to obtain a first porous soot layer; further depositing fine silica particles thereon to obtain a second porous soot layer; subjecting the resulting product to dehydration treatment; vitrifying it into a transparent product and drawing out the heat-resistant base body to obtain a cylindrical quartz glass ingot; and then subjecting the thus obtained quartz glass ingot to a mechanical grinding and the like; and (2) an example of employing VAD method, which comprises forming a soot body by depositing fine silica glass particles generated by flame hydrolysis of high purity silicon tetrachloride on a heat-resistant base body, wherein gases of starting materials differing in composition are supplied separately from a plurality of burners to simultaneously form a plurality of porous soot layers each containing different doping agents; subjecting the resulting product to dehydration treatment; and vitrifying it into a transparent product and thus obtaining a quartz glass tube by subjecting the thus obtained quartz glass ingot to a mechanical grinding and the like. In either of the methods above, it is required that the high temperature viscosity is varied in the wall thickness direction of the quartz glass tube, and that the inner layer side has a lower high temperature viscosity as compared with the outer layer side. In order to accomplish this requirement, it is preferred that a doping agent is doped as described above, and in doping the doping agents, mentioned are, for example, a method comprising supplying the doping agent to the oxyhydrogen flame together with the gaseous starting material on forming the porous soot body, and a method comprising doping the doping agent in any of the production steps starting from the one before or after the dehydration treatment of the porous soot body to step of vitrifying the body into a transparent body. In doping chlorine or fluorine, in particular, the dehydration treatment is performed at the same time of doping. Accordingly, it is effective to subject the porous soot body to a heat treatment in an atmosphere of gaseous chlorine or gaseous fluorine. Furthermore, in case of producing synthetic quartz, there can be mentioned a method of not incorporating a step of obtaining a porous soot body, such as the direct method (DQ process) or sol-gel process. Separately, there also is a method of obtaining a fused glass from a synthetic or a naturally occurring silica powder, but in those production methods or in the aforementioned OVD or VAD methods, a quartz glass tube comprising an inner layer side having a lower high temperature viscosity as compared with the outer layer side can be obtained by preparing, in beforehand, two types of quartz glass tubes differing in high temperature viscosity, and by then melt welding the both tubes into a monolithic body.

In the production of the optical fiber preform using the quartz glass preform for optical fibers, the process comprises carefully inserting the core glass rod for optical fiber preforms into the quartz glass tube for preforms; fixing the core glass rod and the quartz glass tube after aligning their circular centers; straightening the entire body for curving, torsion, etc., preferably, after joining the both ends thereof to a quartz glass dummy tube; inserting the resulting body by its lower end into a vertical electric furnace from the upper side of the furnace; and, sequentially heating in zones in the temperature range of from 1700 to 2000°C to melt weld the resulting body into a monolithic body. The term „sequentially heating in zones" above is the so-called zone melting, and refers to heating in which the heating zone is gradually moved.

The core glass rod for optical fiber preforms for use in the production method above for optical fiber preforms is a light transmitting portion, and mentioned is a quartz glass rod or a quartz glass rod having formed on the periphery thereof an optical clad portion. That is, in the present invention, a „core glass rod" collectively refers to a core rod and to a cladded core rod. A core rod without a clad can be formed by a known VAD process, OVD process, etc., while a cladded core rod can be formed by a method comprising jacketing the core rod with a quartz glass tube, by a method comprising forming a clad portion on the periphery of the core rod by means of OVD process and the like, or by a method comprising a combination of the methods above.

Since the outer surface of the quartz glass tube for preforms above is maintained at a high precision, it is possible to further superpose a quartz glass tube for clads on the outer surface thereof, i.e., to perform the so-called secondary jacketing and tertiary jacketing, and hence, the quartz glass tube for preforms can be used to efficiently produce a further larger optical fiber preforms.

Embodiment of the Present Invention

Embodiment of the present invention is described below, but it should be understood that the present invention is not limited thereto.

Examples

In the Examples and Comparative Examples below, the fluorine concentration and the chlorine concentration are measured by silver nitrate nephelometry, and the concentration of OH groups is measured in accordance with the measuring method described in D.M. Dodd and D.B. Fraser, Optical determination of OH in fused silica, *Journal of Applied Physics*, Vol. 37 (1966).

Example 1

A porous soot body was produced in accordance with OVD method by vaporizing a high purity silicon tetrachloride, performing flame hydrolysis in an oxyhydrogen flame, and depositing the resulting product on the periphery of a base body 50 mm in outer diameter being rotated at a rate of 50 rpm. To the burner for use in the flame hydrolysis above were supplied 1500 g/h of silicon tetrachloride as the starting material, 1.8 m³/h of gaseous hydrogen, and 0.9 m³/h or gaseous oxygen. Thus was obtained a porous soot body about 230 mm in outer diameter and about 3500 mm in length. Thus obtained porous soot body was placed inside an electric furnace, was heated at 1100 °C in an atmosphere of mixed gaseous silicon tetrafluoride and gaseous nitrogen, and was treated subsequently at the same temperature in an atmosphere of mixed gaseous chlorine and gaseous nitrogen.

Porous soot was then deposited on the periphery of the thus obtained porous soot body by means of OVD process, and the resulting product was heated at 1100°C in an atmosphere of mixed gaseous chlorine and gaseous nitrogen. The newly obtained porous soot body had an outer diameter of about 400 mm. The porous soot body thus obtained was vitrified in gaseous nitrogen at a temperature of 1600 °C to obtain a transparent body as a result, from which the base body was drawn out to produce a cylindrical quartz glass ingot. The resulting cylindrical quartz glass ingot had an outer diameter of about 200 mm and an inner diameter of about 50 mm. The both ends of the quartz glass ingot were cut, and thus was produced a quartz glass tube 195 mm in outer diameter and 55 mm in inner diameter by mechanically milling and mechanically polishing the inner and outer peripheral surfaces.

Although slight differences in thickness was observed at the central portion of the wall of the resulting quartz glass tube, no difference in the distribution of refraction index was observed on the outer layer side and the inner layer side over the entire wall thickness. A sample for measurement was cut out from the edge portion of the quartz glass tube, and the concentration for fluorine and chlorine was measured thereon. The fluorine concentration and the chlorine concentration in the inner layer side for a thickness of 35 mm as measured from the inner peripheral surface were found to be 500 ppm and 2000 ppm, respectively. The chlorine concentration for the outer layer side in a region having a thickness of 35 mm as measured from the outer peripheral surface was found to be lower than the detection limit of 30 ppm. The viscosity of the quartz glass tube was measured, and was found to be 1×10^{11} Poise at 1280 °C for the inner layer side and 1×10^{12} Poise at 1280 °C for the outer layer side. The viscosity above are values obtained by cutting out samples $3 \times 3 \times 50$ mm in size from the inner layer side and the

outer layer side, and then measuring by beam bending method, i.e., by supporting each of the cut out samples by two points under a temperature of 1280°C, and by measuring the quantity of deformation caused by its own weight. Further, the residual OH group concentration of the quartz glass tube was found to be 0.1 ppm.

In earlier measurements of samples produced under similar conditions a fluorine and chlorine concentration in the inner layer side (for a thickness of 35 mm) was found to be of 500 ppm and 3000 ppm, respectively. The chlorine concentration for the outer layer side in a region having a thickness of 35 mm as measured from the outer peripheral surface in that experiment was found to be 1000 ppm. The viscosity of the quartz glass tube was measured, and was found to be 1×10^5 Poise at 1280 °C for the inner layer side and $1 \times 10^{17.5}$ Poise at 1280 °C for the outer layer side.

Separately, a cladded core rod for optical fiber preforms was produced by VAD process, which was hot stretched in a vertical electric furnace to obtain a core glass rod 50 mm in outer diameter. The core glass rod was carefully inserted into the quartz glass tube above so that it may not be brought into contact with the inner peripheral surface of the tube, and was fixed to the quartz glass tube after aligning the circular center thereof with that of the quartz glass tube. After joining each of the both ends of the resulting product with a dummy quartz material, the resulting body was placed inside a vertical electric furnace from the lower end, and the lower end was melt welded. The inside of the quartz glass tube was reduced in pressure, and was sequentially heated in zones to obtain a monolithic body by melt welding. The melt welding temperature was 1800 °C. The distribution of the refraction index of the thus obtained optical fiber preform was measured every 50 mm by using a preform analyzer to found a deviation of± 0.2 mm or less with respect to the outer diameter. Furthermore, white light was irradiated to the edge plane in a dark room, but no bubbles 0.1 mm or more in size, i.e., the minimum visually observable unit, were observed.

Example 2

A porous soot body was produced in accordance with OVD method by vaporizing a high purity silicon tetrachloride, performing flame hydrolysis in an oxyhydrogen flame, and depositing the resulting product on the periphery of a base body 50 mm in outer diameter being rotated at a rate of 50 rpm. To the burner for use in the flame hydrolysis above were supplied 1500 g/h of silicon tetrachloride as the starting material, 1.8 m^3/h of gaseous hydrogen, and 0.9 m^3/h or gaseous oxygen. Thus was obtained a porous soot body about 230 mm in outer diameter and about 3500 mm in length. Thus obtained porous soot body was placed inside an electric furna-

ce, was heated at 1100 °C in an atmosphere of mixed gaseous silicon tetrafluoride and gaseous nitrogen, and was treated subsequently at the same temperature in an atmosphere of mixed gaseous chlorine and gaseous nitrogen.

Porous soot was then deposited on the periphery of the thus obtained porous soot body by means of OVD process, and the resulting product was heated at 1100°C in an atmosphere of mixed gaseous chlorine and gaseous nitrogen. The newly obtained porous soot body had an outer diameter of about 400 mm. The porous soot body thus obtained was vitrified in gaseous nitrogen at a temperature of 1600 °C to obtain a transparent body as a result, from which the heat-resistant base body was drawn out to produce a cylindrical quartz glass ingot. The resulting cylindrical quartz glass ingot had an outer diameter of about 200 mm and an inner diameter of about 50 mm. The both ends of the quartz glass ingot were cut, and thus was produced a quartz glass tube 195 mm in outer diameter and 55 mm in inner diameter by mechanically milling and mechanically polishing the inner and outer peripheral surfaces.

Although slight differences in thickness was observed at the central portion of the wall of the resulting quartz glass tube, no difference in the distribution of refraction index was observed on the outer layer side and the inner layer side over the entire wall thickness. A sample for measurement was cut out from the edge portion of the quartz glass tube, and the concentration for fluorine and chlorine was measured thereon. The fluorine concentration and the chlorine concentration in the inner layer side for a thickness of 35 mm as measured from the inner peripheral surface were found to be 500 ppm and 3000 ppm, respectively. The chlorine concentration for the outer layer side in a region having a thickness of 35 mm as measured from the outer peripheral surface was found to be 1000 ppm. The viscosity of the quartz glass tube was measured in accordance with the method similar to that used in Example 1, and was found to be $1 \times 10^{10.5}$ Poise at 1280 °C for the inner layer side and $1 \times 10^{11.5}$ Poise at 1280 °C for the outer layer side. Further, the residual OH group concentration of the quartz glass tube was found to be 0.1 ppm.

Separately, a cladded core rod for optical fiber preforms was produced by VAD process, which was hot stretched in a vertical electric furnace to obtain a core glass rod 50 mm in outer diameter. The core glass rod was carefully inserted into the quartz glass tube above so that it may not be brought into contact with the inner peripheral surface of the tube, and was fixed to the quartz glass tube after aligning the circular center thereof with that of the quartz glass tube. After joining each of the both ends of the resulting product with a dummy quartz material, the resulting body was placed inside a vertical electric furnace from the lower end, and the lower

end was melt welded. The inside of the quartz glass tube was reduced in pressure, and was .
sequentially heated in zones to obtain a monolithic body by melt welding. The melt welding
temperature was 1800 °C. The distribution of the refraction index of the thus obtained optical
fiber preform was measured every 50 mm by using a preform analyzer to found a deviation of±
0.2 mm or less with respect to the outer diameter. Furthermore, white light was irradiated to the
edge plane in a dark room, but no bubbles 0.1 mm or more in size, i.e., the minimum visually
observable unit, were observed.

Effect of the Invention

The quartz glass tube for optical fiber preforms according to the present invention is va-
ried in high temperature viscosity in the wall thickness direction in such a manner that the high
temperature viscosity in the inner layer side is lower than that of the outer layer side. Thus, by
using the quartz glass tube for optical fiber preforms according to the present invention in the
production of optical fiber preforms, the melt welding temperature for obtaining a monolithic bo-
dy with a core glass rod for the optical fiber preform can be lowered as to obtain an optical fiber
preform with high precision while reducing the construction cost of the heating furnace, and yet,
without impairing the working environment. Furthermore, an optical fiber preform having fa-
vorable distribution of refraction index can be obtained without causing the diffusion of doping
agent doped in the core portion.

Claims

1.   A quartz glass tube for use in the production of optical fiber preforms, characterized in that said quartz glass tube shows the high temperature viscosity varying in the wall thickness direction, and that the high temperature viscosity in the inner layer side of the wall of the quartz glass tube is lower than that of the outer layer side.

2.   A quartz glass tube for an optical fiber preform as claimed in Claim 1, wherein the viscosity of the inner layer side at 1280 °C is in the range of from 1×10^4 to 1×10^{12} Poise, and the viscosity of the outer layer side at 1280 °C is in the range of from 1×10^{11} to 1×10^{13} Poise.

3.   A quartz glass tube for an optical fiber preform as claimed in Claim 1 or 2, wherein the variation in high temperature viscosity in the wall thickness direction is set by doping a doping agent.

4.   A quartz glass tube for an optical fiber preform as claimed in any of Claims 1 to 3, wherein at least one of the elements selected from the group consisting of chlorine, germanium, phosphorus, and fluorine is doped in the inner layer side of the wall of the quartz glass tube.

5.   A quartz glass tube for an optical fiber preform as claimed in any of Claims 1 to 4, wherein at least one of the elements selected from the group consisting of chlorine, germanium, and phosphorus is doped together with fluorine in the inner layer side of the quartz glass tube, and chlorine is doped in the outer layer side.

6.   A method for producing an optical fiber preform comprising inserting a core glass rod into a quartz glass tube and the melt weld them together into a monolithic product, characterized in that the high temperature viscosity of said quartz glass tube is varied in the

wall thickness direction, and that the high temperature viscosity of the inner layer side or the quartz glass tube is lower than that of the outer layer side.

7. A method for producing an optical fiber preform as claimed in claim 6, wherein the viscosity of the inner layer side of the quartz glass tube at 1280°C is in the range of from 1×10^4 to 1×10^{12} Poise, and the viscosity of the outer layer side of said quartz glass tube at 1280 °C is in the range of from 1×10^{11} to 1×10^{13} Poise.

8. A method for producing an optical fiber preform as claimed in claim 6 or 7, wherein variation in high temperature viscosity in the wall thickness direction is set by doping with a doping agent.

9. A method for producing an optical fiber preform as claimed in any of claim 6 to 8, wherein at least one of the elements selected from the group consisting of chlorine, germanium, phosphorus, and fluorine is the doping agent in the inner layer side of the wall of the quartz glass tube.

10. A method for producing an optical fiber preform as claimed in any of claim 6 to 9, wherein at least one of the elements selected from the group consisting of chlorine, germanium, and phosphorus is doped together with fluorine in the inner layer side of the quartz glass tube, and chlorine is doped in the outer layer side.

INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7   C03B37/012

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7   C03B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 8, no. 225, 16 October 1984 (1984-10-16) & JP 59 111939 A (HITACHI CABLE CO. LTD.), 28 June 1984 (1984-06-28) abstract ---	1,3,4,6, 8,9
X	PATENT ABSTRACTS OF JAPAN vol. 11, no. 259, 21 August 1987 (1987-08-21) & JP 62 059543 A (MITSUBISHI CABLE IND. LTD.), 16 March 1987 (1987-03-16) abstract --- -/--	1,3,6,8

[X] Further documents are listed in the continuation of box C.   [X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

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Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 3, no. 138, 16 November 1979 (1979-11-16) & JP 54 116425 A (HITACHI CABLE CO. LTD.), 10 September 1979 (1979-09-10) abstract	1,6

X	EP 0 055 822 A (NORTHERN TELECOM LTD.) 14 July 1982 (1982-07-14) claims 9,12-15,17	1,3,4

X	EP 0 520 402 A (SUMITOMO ELECTRIC IND. LTD.) 30 December 1992 (1992-12-30) claims 1,3,5-7,11	1,3,4

X	DE 39 41 864 A (AEG KABEL AG) 20 June 1991 (1991-06-20) claims 1,3-7	1,3,4

X	DE 39 23 686 A (AEG KABEL AG) 24 January 1991 (1991-01-24) claims 1,3,6	1,3,4

X	DATABASE WPI Section Ch, Week 21 Derwent Publications Ltd., London, GB; Class L01, AN 1991-152336 XP002134073 -& JP 03 087020 A (SHIN-ETSU SEKIEI CO. LTD.), 11 April 1991 (1991-04-11) abstract; figure 2	1

X	PATENT ABSTRACTS OF JAPAN vol. 1, no. 80 (E-032), 27 July 1977 (1977-07-27) & JP 52 017037 A (SUMITOMO ELECTRIC IND. LTD.), 8 February 1977 (1977-02-08) abstract	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
JP 59111939	A	28-06-1984	NONE		
JP 62059543	A	16-03-1987	JP	1943982 C	23-06-1995
			JP	6065613 B	24-08-1994
JP 54116425	A	10-09-1979	NONE		
EP 0055822	A	14-07-1982	CA	1170876 A	17-07-1984
			JP	57129836 A	12-08-1982
EP 0520402	A	30-12-1992	JP	5004832 A	14-01-1993
			JP	5105467 A	27-04-1993
			JP	5058664 A	09-03-1993
			JP	5105466 A	27-04-1993
			AU	649845 B	02-06-1994
			AU	1843392 A	07-01-1993
			DE	69215901 D	23-01-1997
			DE	69215901 T	07-05-1997
			DE	69219677 D	19-06-1997
			EP	0626352 A	30-11-1994
			KR	9504059 B	25-04-1995
			US	5314518 A	24-05-1994
DE 3941864	A	20-06-1991	NONE		
DE 3923686	A	24-01-1991	NONE		
JP 03087020	A	11-04-1991	JP	2610056 B	14-05-1997
JP 52017037	A	08-02-1977	JP	1168432 C	30-09-1983
			JP	57061698 B	25-12-1982